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# Influence of Heat Treatment on Ordering Process in Fe<sub>72</sub>Al<sub>28</sub> Alloy Studied by Mössbauer Spectroscopy

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The paper discusses a theoretical model that associates the shape of Mössbauer spectrum with the configuration of atoms in local surroundings of the Mössbauer nuclide. The model has been implemented to a computer program which was used to analyse the Mössbauer spectra of Fe<sub>72</sub>Al<sub>28</sub> alloys after various heat treatments. Basing on the determined configuration of atoms, the long range ordering parameter was estimated.

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## 1. Introduction

It is well established that physical and mechanical properties of Fe-Al alloys are related to their atomic ordering [1]. Mössbauer spectroscopy offers a sensitive microscopic point-probe to identify the nature of <sup>57</sup>Fe-atom configurations responsible for various hyperfine fields observed in Fe-Al alloys [2–3]. The work presents a method of determining the level of long range ordering in the alloys characterized by superstructure D0<sub>3</sub>. This method is a development of the method proposed by the authors of [3]. In the present approach, the possible atomic configurations around Mössbauer probe are the basis for reconstruction of Mössbauer spectrum. The degree of ordering is expressed by a sum of populations of chosen atomic configurations characteristic for entirely ordered structure.

## 2. Experimental

The materials investigated were multicomponent Fe<sub>28</sub>Al alloys of nominal composition 71.64 at.% Fe, 28 at.% Al and small amounts of other additives (Mo — 0.2 at.%, C — 0.1 at.%, Zr — 0.05 at.%, B — 0.01 at.%) introduced in order to improve their thermal and mechanical properties. The alloys were produced by vacuum induction melting. The samples were examined in seven different states defined in Table I.

The X-ray diffraction analysis of the investigated samples indicated the presence of D0<sub>3</sub> (basing on the BCC lattice) depending on the samples' compositions and the kind of heat treatment they underwent (Table I).

Mössbauer spectroscopy studies in the standard transmission geometry were carried out for thin foils (about

TABLE I

The thermal treatment of the investigated samples.

No. of sample		Treatment
1	FC	homogenization 48 h at 1000 °C in air, slow cooling with furnace
2	Q-Air	homogenization 48 h at 1000 °C in air, quenching in air
3	Q-Oil	homogenization 48 h at 1000 °C in air, quenching in oil
4	Q-Air 500° 0.5 h	Sample 2 annealed at 500 °C for 0.5 h / slow cooling with furnace
5	Q-Oil 500° 0.5 h	Sample 3 annealed at 500 °C for 0.5 h / slow cooling with furnace
6	Q-Air 500° 2 h	Sample 4 annealed at 500 °C for 2 h / slow cooling with furnace
7	Q-Oil 500° 2 h	Sample 5 annealed at 500 °C for 2 h / slow cooling with furnace

40 μm thick) of samples at room temperature using a constant acceleration drive. The source was <sup>57</sup>Co(Rh) with an activity of about 10 mCi (one spectrum was measured with source of activity of 50 mCi).

For the numerical analysis of the spectra, specially created software was used which implemented a theoretical model appropriate to solve the problem.

## 3. Theoretical model of spectrum and data analysis

It is assumed that the spectrum can be deconvoluted into a complex of sextets of lines. Each sextet relates to a specific atomic configurations in two nearest neighboring shells of <sup>57</sup>Fe. The influence of the nearest neighboring shells is determined by the number of various atoms in

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those shells, but not by their space arrangement. Taking into account the above assumptions and the fact that for the BCC structure there are 8 sites in the first and 6 sites in the second shell, the cross section  $S(v)$  of the resonant absorption is

$$S(v) = \sum_{m=0}^8 \sum_{n=0}^6 P(m, n) L_6(v, m, n), \quad (1)$$

where  $v$  is the relative velocity of source-absorbent and  $m$  and  $n$  denote the numbers of Al atoms in the nearest and the next nearest shell of  $^{57}Fe$ , respectively.  $P(m, n)$  is the frequency of the appearance of the  $(m, n)$  configuration and  $L_6(v, m, n)$  is the Zeeman sextet relating to this configuration.

The positions of lines in each sextet are calculated via respective hyperfine parameters: the hyperfine magnetic field  $H(m, n)$ , isomer shift  $IS(m, n)$  and quadruple splitting  $QS(m)$  (the latter is assumed to be a function of  $m$  solely). In order to reduce the number of free parameters in Eq. (1), the following constraints have been imposed: according to literature suggestions [4–5],  $H(m, n) = H(0, 0) - m\Delta H_I - n\Delta H_{II}$ , where  $H(0, 0)$ ,  $\Delta H_I$  and  $\Delta H_{II}$  are constant values.  $IS(m, n)$  is calculated as a linear function of the hyperfine field  $IS(m, n) = IS_0 + \beta H(m, n)$ . Such a correlation was observed in literature for mean values of  $H$  and  $IS$  [4, 6] as well as for respective local value of  $H$  and  $IS$  [6–7]. The model assumes also that the hyperfine interactions of farther coordinative shells lead to a broadening of the spectrum lines and that the broadening (in relation to natural width of line  $\Gamma_0$ ) depends on the distance of a given line from the centre of spectrum  $\Delta\Gamma = \delta|v_i|\Gamma_0$ , where  $v_i$  is the  $i$ -th line position.

$P(m, n)$  (Eq. (1)) is approximated as a product  $P(m, n) = p_I(m)p_{II}(n|m)$ , where  $p_I(m)$  is the probability of finding  $m$  atoms of Al in the first coordinative shell of the Mössbauer probe and  $p_{II}(n|m)$  is the conditional probability of finding  $n$  atoms of Al in the second shell given the presence of  $m$  atoms Al in the first shell. For further reduction of free parameters and to obtain smooth changes of  $p_{II}(n|m)$ , the conditional probability is approximated by binomial distribution  $p_{II}(n|m) = C_m^n p_m^n (1 - p_m)^{6-n}$ , where  $p_m$  is a parameter different for different  $m$ .

Finally, the Mössbauer spectrum  $M(v)$  of the investigated alloys is expressed by:

$$M(v) = B - C\varepsilon(v), \quad (2)$$

where  $B$  and  $C$  are constants, and  $\varepsilon(v) = \int_{-\infty}^{\infty} dv' R(v') T(v' - v)$  is the transmission integral of spectrum.  $R(v)$  describes the shape of the emission line, while  $T(v) = 1 - \exp(-t_a S(v))$  depends on the resonant absorption of gamma radiation. The  $t_a$  parameter is the effective thickness of the absorbent.

The theoretical spectrum (Eq. (2)) was fitted to experimental data. All the seven spectra after different heat treatment (Table I) were fitted simultaneously. The

searched values of parameters  $H(0, 0)$ ,  $\Delta H_I$ ,  $\Delta H_{II}$ ,  $IS_0$ ,  $\beta$ ,  $\delta$  and  $QS(m)$  were assumed to be common for all the spectra fitted together.

#### 4. Results and discussion

According to the phase diagram for the Fe-Al system [8], if the concentration of Al is close to 28%, the  $D0_3$  phase is the equilibrium phase. Indeed, the XRD patterns related to all investigated Fe28Al samples indicated the presence of  $D0_3$  phase in various amounts. In Fig. 1, two selected XRD patterns are presented. Although both XRD patterns indicate presence of the  $D0_3$  superstructure, intensities of the characteristic reflexes in the XRD patterns for the Q-oil sample are much lower than the ones for the FC sample.

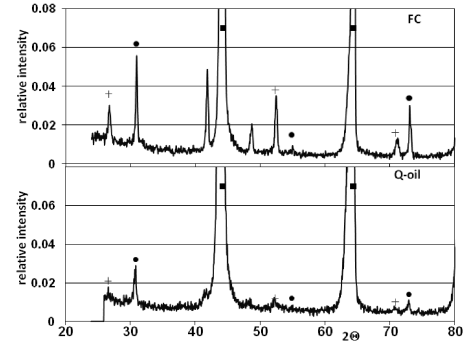


Fig. 1. X-ray diffraction patterns of Fe28Al samples annealed at 1000 °C for 48 hours and 1) cooled slowly with furnace (FC); 2) quenched in oil (Q-oil). The crosses denote peaks related to  $D0_3$  superstructure, circles — to B2 structure and squares — to A2 structure. Other peaks likely relate to precipitations of some minor alloy additives. The intensities are normalized in relative to the height of maximal peak.

TABLE II

The determined values of some hyperfine structure parameters.

$\Delta H_I$ [T]	$\Delta H_{II}$ [T]	$H(0,0)$ [T]	$IS_0$ [mm/s]*	$\beta$ [mm/s T <sup>-1</sup> ]
3.29	1.19	33.6	0.353	-0.00926

\*in relation to  $\alpha$ -Fe standard

Despite the relatively strong constraints imposed on the model parameters, very good fits to the examined Mössbauer spectra were obtained. The determined values of the common parameters are presented in Table II. Figure 2 shows experimental spectra and theoretical fits to the data, and Figs. 3–4 — the distribution of  $P(m, n)$  (Eq. (1)) determined for each spectrum. In the completely ordered structure of  $D0_3$ , there are only two configurations of Al atoms in two first coordination shells of Fe atom, namely configuration  $(m = 4, n = 0)$  with frequency  $P(4, 0) = 2/3$  and configuration  $(m = 0, n = 6)$

with frequency  $P(0,6) = 1/3$ . Fe<sub>28</sub>Al is not, so even in the completely ordered phase, some additional configurations besides the mentioned above have to exist. Figure 3a shows a predicted contribution  $P(m,n)$  for Fe-Al of 28% of Al atoms. The diagram was obtained by computer simulation with an assumption that the whole material is only one entirely ordered crystallite and that the excess of Al atoms is distributed to all sites with the same probability.

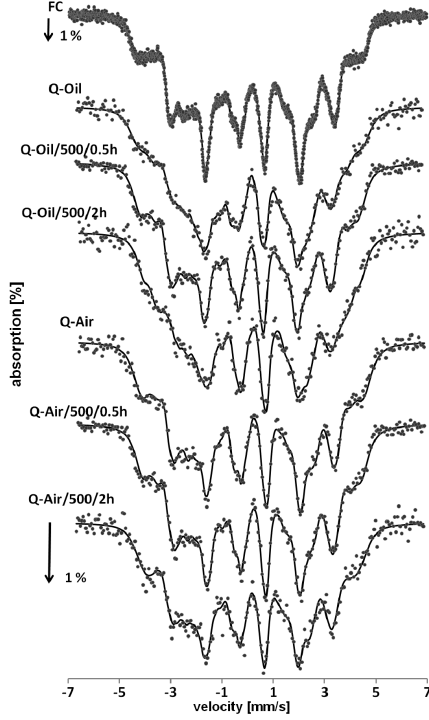


Fig. 2. The Mössbauer spectra (points) and theoretical fits (lines) for Fe<sub>28</sub>Al after different heat treatment. All the spectra were fitted simultaneously with correlations between chosen fitting parameters.

In the case of our samples, the ideally ordered structure is impossible to obtain because the samples are not in the equilibrium state, they have a non-stoichiometric composition and contain defects and antiphase domains. Because of this, one may expect a number of various atomic configurations  $(m,n)$ . Indeed, the experimentally obtained  $P(m,n)$  distributions are quite complex, however almost all of them contain peaks at  $(4,0)$  and  $(0,6)$ . It is especially seen in the diagram of determined  $P(m,n)$  for furnace cooled sample (Fig. 3b).

To precisely determine the level of alloy ordering, one would have to make such a simulation of its structure that would reflect the determined  $P(m,n)$  distribution. Such a procedure is very complex and difficult to be carried out. Below, a simple method is presented, which we hope to be able to determine the ordering level. As a temporary measure of ordering, we propose the parameter  $S$  defined by the below equation:

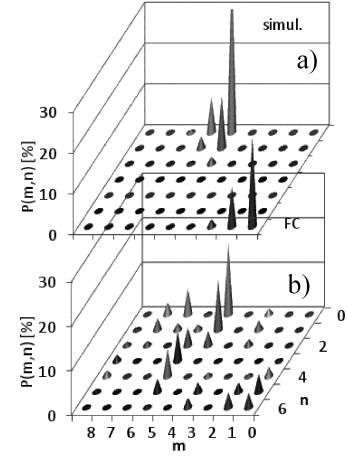


Fig. 3. (a) The simulated distribution of  $P(m,n)$  for completely ordered Fe<sub>28</sub>Al crystallite of D<sub>03</sub> superstructure; (b) The  $P(m,n)$  distribution determined from Mössbauer spectrum of the furnace cooling sample (FC).

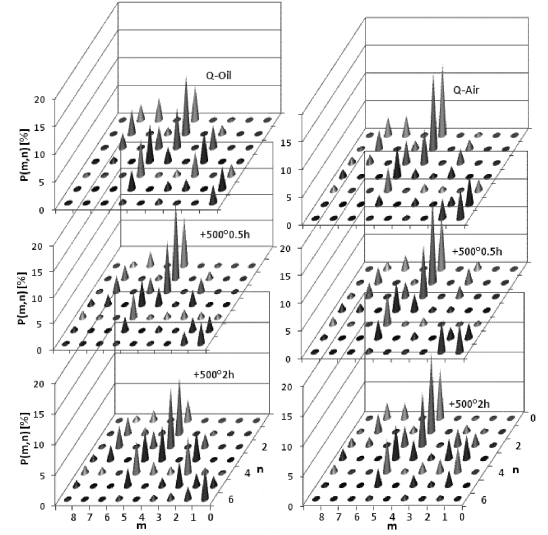


Fig. 4. The  $P(m,n)$  distributions determined from Mössbauer spectra of samples that underwent various heat treatment (see Table I).

$$S = \frac{(P(4,0) + P(4,1) + P(0,6) + P(1,6))}{(P_{\max}(4,0) + P_{\max}(4,1) + P_{\max}(0,6) + P_{\max}(1,6))}. \quad (3)$$

The  $S$  parameter makes use of the populations of configurations  $P(m,n)$  that are characteristic for D<sub>03</sub> structure.  $P_{\max}(m,n)$  denote values of the populations for the completely ordered D<sub>03</sub> structure determined by the modelling (Fig. 3a).

Figure 5 shows the changes of  $S$  calculated on the basis of experimentally determined  $P(m,n)$  distributions for Fe<sub>28</sub>Al samples that underwent different heat treatment.

The highest value of  $S$  parameter was determined for

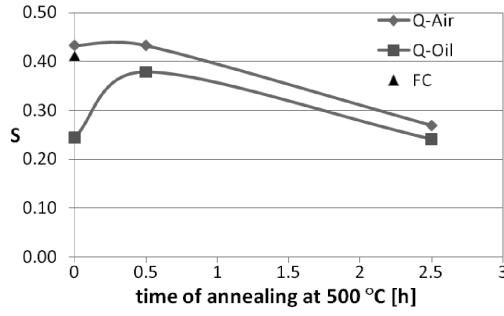


Fig. 5. The changes of  $S$  parameter calculated from Eq. (3) for the investigated samples.

FC and Q-Air samples while  $S$  for Q-Oil sample was much lower. This result seems reasonable since fast quenching to oil should freeze the largest amount of high-temperature phase (B2). Heating the Q-Air sample at 500 °C caused a monotonic drop of  $S$  while heating the Q-Oil sample at the same temperature for 0.5 h leads to an increase in the  $S$  parameter. Further heating decreased  $S$  again. The tendency to lower the degree of the order during annealing at 500 °C is against expectations. According to the phase diagram, one could expect some increase in order. The opposite tendency may be caused by the minor additives in the alloy, which could lower the temperature of phase transformation. Anyhow, such a behavior is confirmed by our XRD results. Indeed, the reflexes of  $D0_3$  superstructure declined with increasing time of annealing of quenched samples at 500 °C.

## 5. Conclusion

The Mössbauer spectroscopy seems to be an effective and promising technique for estimation of degree of order in alloys based on iron. The proposed method of analysis of Mössbauer spectra can give results even more informative than the results of XRD measurements.

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